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10/602755

CL1459 US DIV

Response to Office Action.

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Patent

In the United States Patent and Trademark Office

Application No. 10/602,755

Confirmation No. 9985

Applicant: Zhen Yu Yang

Filed: June 24, 2003

Group Art Unit: 1713

Examiner: Henry Hu

Docket No. CL-1459 US DIV

Customer No. 23906

November 23, 2005

Response to Office Action

Mail Stop Amendment
Commissioner for Patents
P.O. Box 1450
Alexandria, Virginia 22313-1450

Sir:

In reply to Paper No. _____, the October 27, 2005 Notice of Non-Compliant Amendment concerning this application, Applicant respectfully submits the following pursuant to 37 CFR §1.111(a)(2)(D):

Amendments

Please amend this application with respect to the matters set forth on separate pages below concerning the written description and claims:

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In the Written Description:

Please delete the original of each of the following paragraphs, and replace it with a replacement paragraph as indicated:

Paragraph Number	Paragraph Location			
	Beginning at		Ending at	
	Page	Line	Page	Line
001	1	13	1	17
002	1	23	1	34
003	2	22	2	25
004	2	26	2	33
005	2	34	3	6
006	3	7	3	12
007	3	13	3	21
008	4	7	4	11
009	5	3	5	16
010	6	35	7	5
011	9	29	10	11

The replacement paragraph for each of the deleted paragraphs identified above is set forth below in a marked-up version of the original of those paragraphs showing the amendments requested thereto, as required by 37 CFR §1.121(b). In all requested amendments, deletions are shown by strike-through, and additions are shown by underlining.

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Paragraph 001

Monomers of the formula



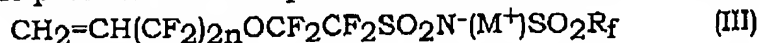
where $n \geq 1$ are disclosed in WO 9831716. $n=1-4$ compositions are explicitly disclosed in Chen et al, "Perfluoro and polyfluorosulfonic acids", Huaxue Xuebao (1982), 40(10), 904-12.

Paragraph 002

See for example, Ezzell et al. U.S. 4,940,525, wherein is used 25 wt % NaOH(aq) for 16 hours at 80-90°C; Banerjee et al. U.S. 5,672,438, wherein is used 25 wt % NaOH for 16 hours at 90°C, or, in the alternative, an aqueous solution of 6-20% alkali metal hydroxide and 5-40% polar organic liquid (e.g., DMSO) for 5 minutes at 50-100°C; Ezzell et al. U.S. 4,358,545 wherein is used 0.05N NaOH for 30 minutes for 50°C; Ezzell et al. U.S. 4,330,654, wherein is used 95% boiling ethanol for 30 minutes followed by addition of equal volume of 30% NaOH (aq) with heating continued for 1 hour; Marshall et al. EP 0345964 A1, wherein is used 32 wt % NaOH (aq) and methanol for 16 hours at 70°C, or, in the alternative, an aqueous solution of 11 wt % KOH and 30 wt % DMSO for 1 hour at 90°C; and, Barnes et al. U.S. 5,595,676, wherein is used 20 wt % NaOH (aq) for 17 hours at 90°C.

Paragraph 003

The present invention provides for a monomer of the formula

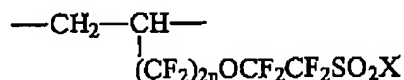


where $n \geq 1$ and $\text{M}^+ = \text{H}^+$ or an alkali metal cation, and R_f is C1-4 perfluoroalkyl optionally substituted by one or more ether oxygens.

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Paragraph 004

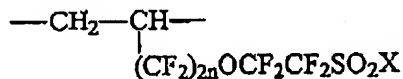
The present invention further provides for a polymer comprising monomer units of VF₂ and 1 to 40 mol % of ionic monomer units of the formula



where $n \geq 1$, X is O⁻M⁺, or N⁻(M⁺)SO₂R_f where M⁺ is H⁺ or an alkali metal cation and R_f is C1-4 perfluoroalkyl optionally substituted by one or more ether oxygens.

Paragraph 005

Further provided is a polymer comprising monomer units of ethylene, tetrafluoroethylene, and 4 to 20 mol % of functionalized monomer units of the formula



where $n \geq 1$, X is F, O⁻M⁺, or N⁻(M⁺)SO₂R_f where M⁺ is H⁺ or an alkali metal cation and R_f is C1-4 perfluoroalkyl optionally substituted by one or more ether oxygens.

Paragraph 006

Further provided is a process for forming a composition of the formula CH₂=CH(CF₂)_{2n}OCF₂CF₂SO₃⁻M⁺ where $n \geq 1$, M⁺ is H⁺ or an alkali metal cation, the process consisting essentially of contacting a composition represented by the formula CH₂=CH(CF₂)_{2n}OCF₂CF₂SO₂F with weakly basic solution of an alkali metal salt or hydroxide in a polar

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solvent, the solution having a pH of less than ca. 12, at a temperature in the range of 0-50°C.

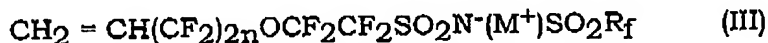
Paragraph 007

Further provided is a process for forming a composition of the formula $\text{CH}_2=\text{CH}(\text{CF}_2)_{2n}\text{OCF}_2\text{CF}_2\text{SO}_2\text{N}^-(\text{K}^+)\text{SO}_2\text{R}_f$ where $n \geq 1$, R_f is C1-4 perfluoroalkyl optionally substituted by one or more ether oxygens, the process consisting essentially of

- forming a 0.001-5 molar solution of $\text{R}_f\text{SO}_2\text{NH}_2$ in an organic solvent;
- combining said solution with $\text{CH}(\text{CF}_2)_{2n}\text{OCF}_2\text{CF}_2\text{SO}_2\text{F}$ and KF to form a mixture;
- heating said mixture to 50-180°C; and
- separating the product.

Paragraph 008

The present invention provides for a monomer represented by the formula

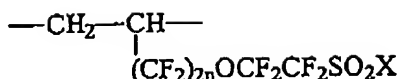


where $n \geq 1$, $n \geq 1$ and $\text{M}^+ = \text{H}^+$ or an alkali metal cation, and R_f is C1-4 perfluoroalkyl optionally substituted by one or more ether oxygens. Preferably R_f is CF_3 , and M^+ is H^+ or Li^+ .

Paragraph 009

The composition of the polymer depends on the ratio of monomers. This was true for all three monomers. One of skill in the art will appreciate that specific reactivity ratios of monomers is determined by the particulars of monomer structure. Accordingly, the present invention provides for an ionomer comprising monomer units of VF_2 and 1 to 40 mol % of monomer units described by the formula

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where $n \geq 1$, $n \leq 1$, X is O^-M^+ , or $\text{N}^-(\text{M}^+)\text{SO}_2\text{R}_f$, where M^+ is H^+ or an alkali metal cation and R_f is C1-4 perfluoroalkyl optionally substituted by one or more ether oxygens. Preferably the concentration of ionic monomer units is 4-20 mol %, most preferably 6-16 mol %. Preferably X is $\text{N}^-(\text{M}^+)\text{SO}_2\text{R}_f$ where M is lithium and R_f is CF_3 .

Paragraph 010

In a preferred embodiment of the process of the invention, monomer (I) and the polymers of the invention formed from (I) are contacted at a temperature in the range of 50-180°C, preferably 70-120°C, with a 0.001-5.0 molar solution of $\text{CF}_3\text{SO}_2\text{NH}_2$ in an organic solvent in the presence of KF precharged to the reaction vessel to form the potassium imide form of (III) or the polymer formed therefrom. Suitable organic solvents include toluene, chlorobenzene, THF, and oligo ethers. Preferred is acetonitrile. Other ionic forms can be formed by contacting the potassium imide form with an alkali metal salt solution, such as LiCl in methanol, or an acid such as aqueous HCl.

Paragraph 011

EXAMPLE 8

Copolymerization of $\text{CH}_2=\text{CHCF}_2\text{CF}_2\text{OCF}_2\text{CF}_2\text{SO}_2\text{F}$ with

TFE and ethylene in F113

A 240-mL stainless steel tube was charged with 100 mL of 1,1,2-trichlorotrifluoroethane (F113), 10 g of $\text{CH}_2=\text{CHCF}_2\text{CF}_2\text{OCF}_2\text{CF}_2\text{SO}_2\text{F}$ and 0.8 g of Lupersol 11 and attached to a gas manifold. The tube was cooled in dry ice and the contents degassed by several cycles of evacuation and repressurization with nitrogen gas. After the final evacuation step, the tube was pressurized with 10 g of ethylene and 30 g of TFE. The tube was then sealed and heated to 60°C and held for

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8 hours to effect polymerization. After completion of the polymerization, the unreacted ethylene and TFE were removed by venting and the white solid was washed with MeOH and dried in a partial vacuum oven at 80°C to give 47.0 g of polymer. IR(KBr): 1464 cm^{-1} (SO_2F). Elementary analysis of polymer indicated that polymer composition was 8.67 parts (CF_2CF_2) and 5.36 parts (CH_2CH_2) to 1 part ($\text{CH}_2\text{CHCF}_2\text{CF}_2\text{OCF}_2\text{CF}_2\text{SO}_2\text{F}$) on a molar basis, based on 37.0% of C, 3.12% of H, 52.3% of F and 2.73% of S. DSC showed that the polymer had T_m of 214°C. By TGA, 10% weight loss was 430°C by TGA in N_2 . A clear transparent and tough film was pressed by placing a sample of the polymer so formed between the platens of a hydraulic press and heated to 250°C with a ram force 20,000 lbs.